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Comparison Study of transition temperature Between the Superconducting Compounds $Tl_{0.9}Pb_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$, $Tl_{0.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$ and $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$

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Abstract

Three high temperature superconductors namely $Tl_{0.9}Pb_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$, $Tl_{0.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$ and $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$ have been successfully prepared by three-step solid state reaction method. The sintering was within (1128-1133) K. Electrical resistively, using four probe techniques, is used to find the transition temperature T_c. The transition temperature at zero resistivity $T_{c(offset)}$ were 110, 115 and 118K, onset superconducting transition temperature $T_{c(onset)}$ were 128, 131 and 132 K for $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$, $Tl_{0.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$ and $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$ respectively. All samples preparation with O_2 flow. X-ray diffraction (XRD) analysis showed a pesudotetragonal structure with changes of lattice parameters for these samples.

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Keywords: Superconductor, Transition temperature, X-ray diffraction and Electrical resistivity

1. Introduction

Tl-based in the form of $TlBa_2Ca_{n-1}Cu_nO_y$ represents the most interesting homologues series out of all known high temperature superconductors. Undoubtedly the primary reason for this is the high critical transition temperature exhibited by this series. Thus the phase with $n = 3$ Tl-1223 exhibits the highest ambient condition critical temperature of ($T_{c(offset)}$ =105–118 K depending on the exact composition, the phase with $n = 2$, Tl- 1212 exhibits the critical temperature $T_{c(offset)}$ =70-105 K) .The grains of these superconductors grow preferentially in the a-b crystallographic directions leading to a high degree of structural anisotropy(1, 2). Tl-1223 superconductor has an irreversibility line located at a relatively high position in $\rho - T$ space, which is due to the relatively short distance between Cu–O layers in the crystal

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lattice, compared to those of other high T_c superconductors. Compared to the Tl-1223 phase and the Tl-1212 phase exhibits a wider processing window and extended phase stability. This makes this material an interesting superconducting compound for both bulk phase applications and for superconducting films(3, 4). Synthesis of these superconductors as single phases requires critical adjustments of the chemicals and the processing conditions (5, 6). Difficulties in the preparation of the undoped compound with high phase purity and some reluctance to work with thallium compounds limited the research on this superconducting compound. Therefore, partial replacement of Tl^{3+} by Pb, Sb or Cr^{3+} (7-9) improves stability , makes the synthesis of single phase material readily feasible and induces a partially melted liquid phase that eases the diffusion of the elements to form the high-T_c phases and to increase their critical current (I_c) (10). we have investigated the Comparison the Superconducting properties Between the Superconducting Compounds $Tl_{0.9}Pb_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$, $Tl_{0.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{9-}$ and $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$. we study of transition temperature, analyze the structure and electrical properties of these superconductors synthesized at the optimum conditions.

2. Experimental

The synthesis of $Tl_{0.9}Pb_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$, $Tl_{0.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$ and $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$ High Temperature Superconductors (HTSC) phases have been prepared by solid state reaction method , using appropriate weights of pure powders (99.998% from May & Baker LTD Dagenham England) materials of Tl_2O_3, Cr_2O_3, Pb_2O_3, Sb_2O_3, BaO_2, CaO and CuO, as starting materials,according to the general chemical formula:

$$(0.45)\ Tl_2O_3 + 0.05\{\ Pb_2O_3,\ Sb_2O_3 \text{ or } Cr_2O_3\ \} + 2\ BaO_2 + 2\ CaO + 3CuO \rightarrow Tl_{0.9}(pb,\ Sb \text{ or } Cr)_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$$

The weight of each reactant was measured by using a sensitive balance type (Mettler H35 AR with Capacity: 110 grams and Readability: 0. 001).The synthesis of the samples have been carried out by three step precursor method. In the first step, the powders (BaO_2, CaO and CuO)were mixed together by using agate mortar ; a sufficient quantity of 2- propane was to homogenization the mixture and to form slurry during the process of grinding for about (30-50) minute. The mixture was dried for an oven at (523K). The mixture was put in furnace that has programmable controller, the powder was heated to temperature of (1073K) for three hours with a rate of (100K/hr) , then cooled to room temperature by the same rate of heating. In the second step, the $Ba_2Ca_2Cu_3O_7$ precursor was mixed with Cr_2O_3 .Pb_2O_3, Sb_2O_3 and Tl_2O_3 to obtain the nominal compositions $Tl_{0.9}\ Pb_{\ 0.1}Ba_2Ca_2Cu_3O_{9-\delta}$, $Tl_{0.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$ and $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$. The powder was pressed into disc-shaped pellets (1.2 cm) in diameter and (0.2-0.3 cm) thick, using hydraulic press type under a pressure of 7 Ton/cm^2.The pellets were presintered in air at (1128-1133) K for (6 hours) with a rate of (200 K/hr) and then cooled to room temperature by same rate of heating.

In the third step, the pellets were reground, repressed and resintered in the oxygen (oxygen rate 0.6 L/min) at the same range of temperature for further (8 hours) and then cooled to (723 K) and annealed in oxygen for (3 hours) and then cooled to room temperature by the same rate of heating.

The samples were examined with resistivity experiments by using standard four-probe technique it is most common method of determining the T_c of a superconductor as shown in figure 1. The sample was fixed in the cryostat instrument which was joined to a rotary pump to get a pressure of 10^{-2} mbar inside the cryostat, and also joined to a sensor of digital thermometer (type Pt 100 resistance to temperature detection RTD) near the sample position. A current 10 mA was supplied to the sample by a current source D.C power supply type (Electronica- Veneta DV 30/V3); the voltage drop was measured by a Keithley model 180 nanovoltmeter with sensitivity of a bout ± 0.1 nanovolt was used for voltage measurements.

The resistivity (ρ) could be found from the relation: $\rho = \dfrac{V}{I}\dfrac{\omega t}{L}$ Where I is the current passing through the sample, V is the voltage drop across the electrodes, ω is the width of the sample, L is the effective length between the electrodes, t is the thickness of the sample. All measurement of L, t and ω

were made by using digital vernier. The excess of oxygen content (δ) could be determine by used chemical method called Iodometric titration.The structure of the prepared sample was obtained by using x-ray diffractometer (XRD) type (Philips) have the following features, the source $Cu_{k\alpha}$ current (20 mA), voltage (40 KV) and λ=1.5405 A^0. A computer program was established to calculate the lattice parameters a,b,c this program based on Cohen's least square method (11).The volume fraction of any phase (V_{phase}) in the sample were determined by using the relation: V_{phase}=

$$\frac{\sum Ia}{\sum I1+\sum I2+......+\sum In}\text{x100}$$

Where *Ia* is the XRD peak intensity of the phase which were determined,$I_1, I_2, ...I_n$ are the peaks intensity of all XRD.

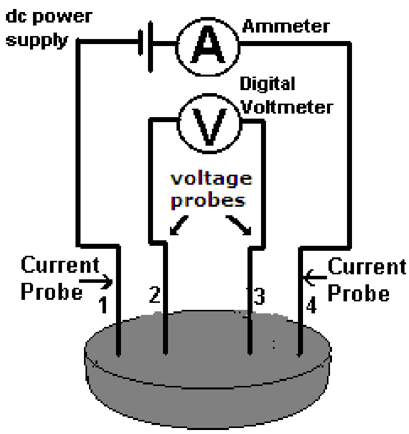

Figure 1. Four–point probe (d.c.) as shown in the figure was used to measure the resistivity (←, at a temperature range (75–300) K, and to determine the critical temperature (T_c).

A computer program was established to calculate the lattice parameters a,b,c this program based on Cohen's least square method .

3. Results and discussion

Resistivity of $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$ (sample A) was measured in the temperature range from 75 K to 300 K and the data are presented in figure 2.

The data points shown in triangle in figure 2, obtained when the sample A was cooling, exhibit an onset superconducting transition temperature $T_{c(onset)}$, of approximately 128 K and offset superconducting transition temperature $T_{c(offset)}$ of 110 K. The superconducting transition width of the sample ΔT_c was found to be 18 K.$Tl_{0.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$ (sample B) possesses an onset superconducting transition temperature T_c, approximately 131 K and offset superconducting transition temperature $T_{c(offset)}$ was 113 K. The superconducting transition width of the sample ΔT_c was found to be 18 K. It is clear that those samples (A and B) also show a broad superconducting transition and this may be due to the presence of impurities or non superconducting regions or multi superconducting phases in the samples.

$Tl_{0.9}Pb_{0.1}Ba_2Ca_2Cu_3O_{9-\delta}$ (sample C) shows a sharp drop with a small transition width in its superconducting transition (14K) and exhibits onset superconducting transition $T_{c(onset)}$, approximately 132 K and offset superconducting transition temperature $T_{c(offset)}$ was 118 K. The sharp drop with a small width in the superconducting transition of this sample indicates that mainly, there is only one superconducting phase Tl(Pb)- 1223 in the sample. However, the small width in the transition indicates that a very small admixture of some other phases exists. Also small differences observed in these data and this may be due to an electronic problem in the experiment.

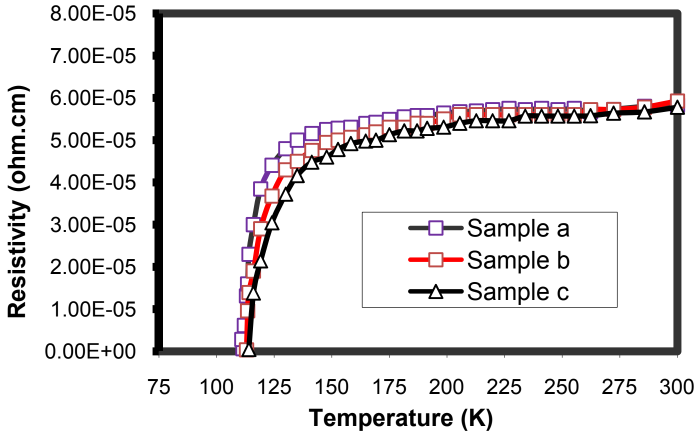

Figure 2. The resistivity dependence on Temperature for a) $Tl_{0.9}Pb_{0.1}Ba_2Ca_2Cu_3O_{8.81}$, b)$Tl_{0.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{8.77}$ and c) $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{8.83}$

According to the X-ray diffraction data the samples consisted of almost phase-pure polycrystalline Tl-1223 phase. The representative XRD patterns are shown in figure 3.

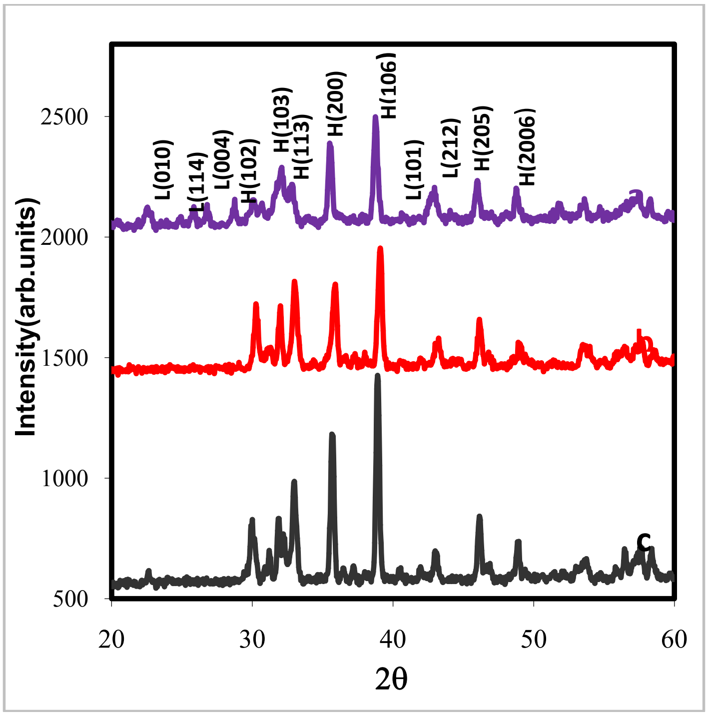

Figure 3. XRD Patterns for the samples a)$Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{8.83}$, b)$Tl_{0.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{8.81}$, and c) $Tl_{0.9}Pb_{0.1}Ba_2Ca_2Cu_3O_{8.77}$

It could be seen from the spectra that there were two main phases in all samples of the Tl-base systems, high-T_c phase (1223), low-T_c phase(1212) and a small amount of impurity phases of Ca_3TlO_6 and Ca_2CuO_3. The appearance of more than two phases could be related to the stacking faults along the c-axis(8). The comparison between the relative intensities of XRD patterns for the samples A,B and C, with the relative intensity shows that all the samples have reflection intensity of the High-T_c phase reflections (peaks H), and Low –T_c phase reflections (peaks L). The high-T_c phase reflections of the sample A has lower intensity than samples B and C.
The formation of the Tl-1223 phase (79.11 % Tl-1223) was observed in the sample $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{8.83}$. The high phase Tl-1223 (85.93%) was obtained for the sample $Tl_{0.9}$ Pb $_{0.1}Ba_2Ca_2Cu_3O_{8.81}$, while the sample $Tl_{0.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{8.77}$ was obtained 81.56% from Tl-1223 phase. The values of transition temperature, oxygen content (δ), lattice parameters a, b, , c/a, Mass density ρ_m and volume fraction (V_{phase}) for all samples are shown in table 1.

Table 1. Values of lattice parameter, C/a, oxygen content (δ) and ρ_M for the samples $Tl_{0.9}Pb_{0.1}Ba_2Ca_2Cu_3O_{8.83}$, $Tl_{0.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{8.81}$, and $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{8.77}$

Samples	$T_{c(On)}$ (K)	(o_2)	a(A^0)	b(A^0)	c(A^0)	*C/a*	ρ_M (g/cm^3)	$V_{Ph-1223}$
$Tl_{0.9}Pb_{0.1}Ba_2Ca_2Cu_3O_{8.83}$	132	0.19	3.831	3.832	15.88	4.145	5.532	85.93
$Tl_{0.9,}$ $Sb_{0.1}Ba_2Ca_2Cu_3O_{8.81}$	131	0.23	3.834	3.836	15.90	4.147	5.556	81.56
$Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{8.77}$	128	0.17	3.833	3.831	15.91	4.153	5.553	79.11

4. Conclusions

In the present study, we have investigated the comparison the superconducting properties Between the Superconducting compounds $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{8.83}$,$Tl_{0.9}$ Pb $_{0.1}Ba_2Ca_2Cu_3O_{8.81}$, and $Tl_{0.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{8.77}$.The as grown samples are dominantly Tl-1223 phases. The transition temperature of as grown samples is found to be sensitive to the Pb on the $Tl_{0.9}$ Pb $_{0.1}Ba_2Ca_2Cu_3O_{8.81}$ compound as comparison with $Tl_{0.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{8.77}$ and $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{8.83}$compounds, it has been observed that maximum $T_{c(offset)}$ was 118 K and $T_{c(onset)}$ was 132 K. It was found that volume fraction $V_{Ph(1223)}$ in the $Tl_{0.9}$ Pb $_{0.1}Ba_2Ca_2Cu_3O_{9-\ \delta}$ compound larger than$_{.9}Sb_{0.1}Ba_2Ca_2Cu_3O_{9-}$ and $Tl_{0.9}Cr_{0.1}Ba_2Ca_2Cu_3O_{9-\delta,}$ change of the Pb, Sb and Cr elements of our samples produce a change in the lattice parameters , volume fraction and mass density $\rho_{m.}$

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